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(54) Title of Invention

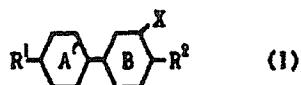
Fluorocyclohexene Derivatives

(57) Abstract

Constitution

Compounds represented by general formula (I)

[Chemical formula 1]



(R<sup>1</sup> is a C<sub>1</sub>-C<sub>16</sub> alkyl group or alkoxyalkyl group; ring A is 2-fluoro-1-cyclohexylene; ring B is phenylene or cyclohexylene; X is F or H; R<sup>2</sup> is F, Cl, CN, C<sub>1</sub>-C<sub>16</sub> alkyl group, alkoxyalkyl group, or alkoxyl group) and liquid crystal compositions that contain these.

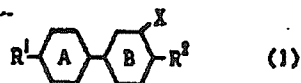
Effect

By adding these compounds, a liquid crystal composition that can be driven at low voltages is obtained, and the refractive index anisotropy of the liquid crystal composition obtained can be made smaller, so application for liquid crystal display elements with a wide-angle field of view is possible.

## CLAIMS

### 1. Compounds represented by general formula (I)

[Chemical formula 1]



(R<sup>1</sup> represents a C<sub>1</sub>-C<sub>16</sub> alkyl group or alkoxyalkyl group; ring A represents a 2-fluoro-1-cyclohexen-1,4-diyl group; ring B represents a 1,4-phenylene group or *trans*-1,4-cyclohexylene group; X represents a fluorine atom or hydrogen atom; and R<sup>2</sup> represents a fluorine atom, chlorine atom, cyano group, C<sub>1</sub>-C<sub>16</sub> alkyl group, alkoxyalkyl group, or alkoxy group, and any hydrogen atom in these groups may be substituted by a fluorine atom).

2. Liquid crystal compositions that contain the compounds represented by general formula (I) of Claim 1.

## DETAILED DESCRIPTION OF THE INVENTION

[0001]

### Industrial field of use

The present invention relates to novel compounds with a fluorocyclohexene ring that are useful as electro-optical liquid crystal display materials.

[0002]

### Prior art

Liquid crystal display elements are used for clocks and calculators as well as various measuring instruments, automotive panels, word processors, personal digital assistants, printers, computers, videos, and the like. As typical liquid display systems, TN (twisted nematic) type, STN (super twisted nematic) type, DS (active light-scattering) type, GH (guest-host) type, and FLC (ferroelectric liquid crystals) are known, but TN and STN are the types most often used at present. Also, as the drive system, multiplex driving is more common than the conventional static driving, and a still simpler matrix system, the active matrix system, has recently been put to practical use.

[0003]

As the liquid crystal materials used in these, a plural number of compounds have thus far been synthesized and are known, but compounds that by themselves satisfy the various properties demanded according to these display systems and applications are not known. The actual situation is that a plural number of compounds are mixed in accord with these demanded properties and are used as liquid crystal compositions. Therefore, the development of liquid crystal compounds that are outstanding in certain properties is extremely important.

[0004]

Liquid crystal compounds usually have as their basic backbone a structure with two or more ring structures (benzene ring or its substituted forms, cyclohexane ring, or a heterocycle such as a pyridine ring, pyrimidine ring, or dioxane ring) bonded directly or via a coupling group. Of these ring structures, many of the compounds with the cyclohexane ring have low viscosity, so they are often used for liquid crystal materials that require high-speed response.

[0005]

In addition, the cyclohexene ring has also been introduced recently. Liquid crystal compounds with a cyclohexene ring are characterized by low viscosity and small refractive index anisotropy ( $\Delta n$ ), and separation of the cis and trans forms is not necessary as in the case of the cyclohexane ring.

[0006]

On the other hand, side substituents such as halogen atoms are being introduced into such ring structures, and the dielectric anisotropy ( $\Delta\epsilon$ ), refractive index anisotropy ( $\Delta n$ ), and elastic constant ( $K$ ) of liquid crystal compounds are also being adjusted. However, this introduction of side substituents is mainly limited to aromatic rings such as the benzene ring, and introduction into the above-mentioned cyclohexene ring has scarcely been investigated because the chemical stability of the product is poor, synthesis is difficult, or other reasons.

[0007]

Problems the invention is to solve

Among the various properties of liquid crystal materials, the elastic constant ( $K$ ) is an extremely important physical property. For any of the various display systems and drive systems mentioned above, as low a threshold voltage ( $V_{th}$ ) as possible is demanded. Usually the threshold

voltage is represented by equation (1)

[0008]

[Mathematical equation 1]

$$V_{th} = k \sqrt{\frac{K}{\Delta \epsilon}} \quad (1)$$

[0009]

(wherein k represents a proportional constant), but as will be recognized from this equation, in order to lower this threshold voltage, it is necessary to make the elastic constant smaller or to make the dielectric anisotropy larger.

[0010]

However, liquid crystal compounds with large dielectric anisotropy are often compounds with high viscosity and are often not suitable for high-speed response. Also, high specific resistance is hardly obtained, so they are difficult to use for active matrix drives. Therefore, compounds with a small elastic constant are necessary for use with such objectives.

[0011]

Also, with the increased capacity of displays, multiplex driving has become necessary, and for that reason, wide-angle viewing is important. For this objective, liquid crystal materials with small refractive index anisotropy ( $\Delta n$ ) are necessary.

[0012]

The problem which this invention is to solve is to provide compounds with a small elastic constant and small refractive index anisotropy in accord with the above objectives and also liquid crystal compositions that contain these compounds.

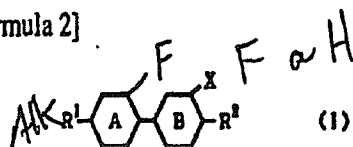
[0013]

#### Means of solving the problems

In order to solve the problems mentioned above, this invention provides fluorocyclohexene derivatives represented by general formula (1)

[0014]

[Chemical formula 2]



[0015]

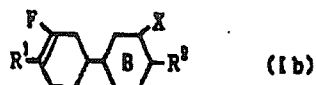
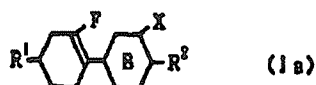
(wherein  $R^1$  represents a  $C_1$ - $C_{16}$  alkyl group or alkoxyalkyl group, but preferably represents a  $C_1$ - $C_8$  straight-chain alkyl group; ring A represents a 2-fluoro-1-cyclohexen-1,4-diyl group; ring B represents a 1,4-phenylene group or *trans*-1,4-cyclohexylene group; X represents a fluorine atom or hydrogen atom, but when ring B represents a *trans*-1,4-cyclohexylene group, X preferably represents a hydrogen atom; and  $R^2$  represents a fluorine atom, chlorine atom, cyano group,  $C_1$ - $C_{16}$  alkyl group, alkoxyalkyl group, or alkoxyl group, and any hydrogen atom in these groups may be substituted by a fluorine atom, but it preferably represents a  $C_1$ - $C_8$  straight-chain alkyl group, alkoxyl group, trifluoromethyl group, trifluoromethoxy group, or fluorine atom).

[0016]

The compounds represented by general formula (I) of this invention can be classified according to the location of the double bond in the cyclohexene ring into general formulas (Ia) and (Ib)

[0017]

[Chemical formula 3]



[0018]

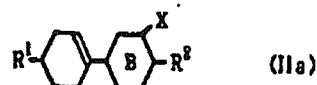
(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in general formula (I)). They can be produced, for example, from the corresponding cyclohexene derivatives according to the method of production shown below.

[0019]

A cyclohexene derivative represented by general formula (IIa)

[0020]

[Chemical formula 4]

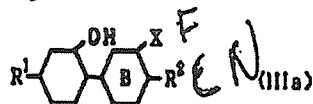


[0021]

(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in general formula (I)) is converted by hydroboration into a cyclohexanol derivative represented by general formula (IIIa)

[0022]

[Chemical formula 5]



[0023]

(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in general formula (I)).

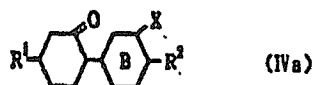
[0024]

Next, this is oxidized to give a cyclohexanone derivative represented by general formula

(IVa)

[0025]

[Chemical formula 6]



[0026]

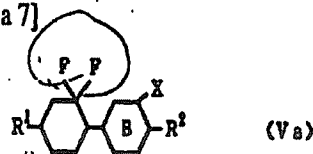
(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in practical example (I)). This is a mixture of cis and trans forms.

[0027]

Next, by fluorinating this with a fluorinating agent such as diethylaminosulfur trifluoride (DAST), a compound of general formula (I) above can be obtained. In this case, 2,2-difluoro-cyclohexane derivatives represented by general formula (Va)

[0028]

[Chemical formula 7]



[0029]

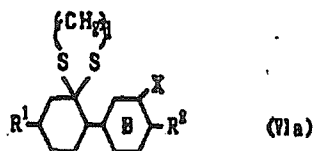
(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in general formula (I)) are also produced. It is thought that compounds of general formula (Ia) are produced from the cis form of the compounds of general formula (IVa) and that compounds of general formula (Va) are produced from the trans form.

[0030]

The fluorination of the cyclohexanone derivatives of general formula (IVa), depending on the compound, is sometimes an extremely slow reaction or does not proceed. In those cases, dithiol is used to give a thioketal derivative represented by general formula (VIa)

[0031]

[Chemical formula 8]



[0032]

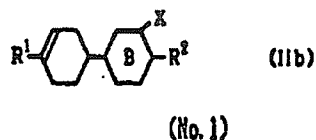
(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in general formula (I)). By reacting this with a fluorinating agent such as a pyridine-hydrogen fluoride complex in the presence of an oxidizing agent such as N-iodosuccinimide (NIS), the difluorocyclohexane derivatives of general formula (Va) can be obtained. If  $R^2$  is an alkoxyl group, its ortho position is sometimes halogenated at this time by iodination, etc. But in that case, the fluorocyclohexene derivatives of general formula (Ia) can be obtained by lithiating with an alkyllithium and then dehalogenating by reacting with water.

[0033]

Among the compounds represented by general formula (I), the fluorocyclohexene derivatives of general formula (Ib) can be obtained from cyclohexene derivatives represented by general formula (IIb)

[0034]

[Chemical formula 9]



[0035]

(wherein  $R^1$ , ring B, X, and  $R^2$  represent the same meanings as in general formula (I)) in exactly the same way as those of general formula (Ia).

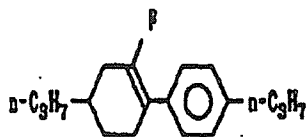


[0036]

A typical example of the compounds represented by general formula (I) produced in this way is shown below.

[0037]

[Chemical formula 10]



[0038]

The compounds represented by general formula (I) of this invention can be used with the aim of lowering the threshold voltage of liquid crystal compositions as, for example, the materials of field-effect display cells of the TN or STN type in particular in the state of a mixture with other nematic liquid crystal compounds that have positive or negative dielectric anisotropy.

[0039]

As the preferred typical examples of nematic liquid crystal compounds that can be mixed with the compounds represented by general formula (I), the following can be mentioned: 4-substituted benzoic acid 4-substituted phenyl ester, 4-substituted cyclohexanecarboxylic acid 4-substituted phenyl ester, 4-substituted cyclohexanecarboxylic acid 4'-substituted biphenyl ester, 4-(4-substituted cyclohexanecarbonyloxy)benzoic acid 4-substituted phenyl ester, 4-(4-substituted cyclohexyl)benzoic acid 4-substituted phenyl ester, 4-(4-substituted cyclohexyl)benzoic acid 4-substituted cyclohexyl ester, 4,4'-substituted biphenyl, 1-(4-substituted phenyl)-4-substituted cyclohexane, 4,4''-substituted terphenyl, 1-(4'-substituted biphenyl)-4-substituted cyclohexane, 2-(4-substituted phenyl)-5-substituted pyrimidine, and 2-(4'-substituted biphenyl)-5-substituted pyrimidine.

[0040]

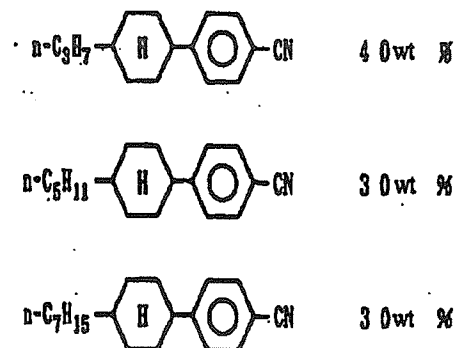
The effects of the compounds of general formula (I) of this invention will be shown by the practical examples described below, but they are also clear from the following examples.

When parent liquid crystals (A) of the phenylcyclohexane system commonly used at present as nematic liquid crystal materials

[0041]

[Chemical formula 11]

Colorless oily substance



[0042]

(wherein the cyclohexane ring represents the trans configuration) were prepared, they showed a nematic phase at  $\leq 50.5^\circ\text{C}$ , and the dielectric anisotropy ( $\Delta\epsilon$ ) was 12.0, the refractive index anisotropy ( $\Delta n$ ) was 0.117, and the threshold voltage ( $V_{th}$ ) of TN cells produced using these was 1.50 V.

[0043]

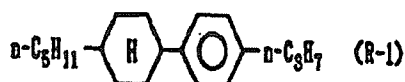
A liquid crystal composition (M-1) comprising 90 wt% of these parent liquid crystals (A) and 10 wt% of compound No. 1 above was prepared. The upper-limit temperature of the nematic phase of this liquid crystal composition (M-1) dropped slightly to  $40.67^\circ\text{C}$ , but its  $\Delta\epsilon$  was 9.8 and its  $\Delta n$  was 0.107, both of which were considerably lower. When cells were produced in the same manner as indicated above and their  $V_{th}$  was determined, it was much lower, 1.26 V, in spite of the fact that  $\Delta\epsilon$  had decreased. Judging from equation (1) above, this shows that the elastic constant (K) of compound No. 1 is extremely low.

[0044]

By contrast, liquid crystal composition (N-1) comprising 10 wt% of a compound of formula (R-1), a compound that has been used for similar objectives in the past,

[0045]

[Chemical formula 12]



[0046]

(wherein the cyclohexane ring represent the trans configuration) and 90 wt% of parent liquid crystals (A) has a nematic phase upper-limit temperature of 39.9°C, virtually unchanged from that of liquid crystal composition (M-1), but its  $\Delta\epsilon$  was 11.3 and its  $\Delta n$  was 0.109, both of which were larger than those of liquid crystal composition (M-1). Moreover, when cells were prepared in the same way as described above and  $V_{th}$  was determined,  $V_{th}$  conversely showed a higher value, 1.37 V, in spite of the fact that  $\Delta\epsilon$  was larger than that of liquid crystal composition (M-1).

[0047]

It is clear from these results that adding a small amount of the compounds represented by general formula (I) of this invention to parent crystals (A) can effectively lower the threshold voltage ( $V_{th}$ ) of the parent liquid crystals and also has the effect of lowering  $\Delta n$ .

[0048]

#### Practical examples

Practical examples of this invention are shown below to further explain the invention. However, this invention is not limited to these practical examples.

[0049]

The phase transition temperature was determined using both a polarizing microscope equipped with a temperature-controlled stage and a differential scanning calorimeter (DSC). The structures of the compounds were confirmed by nuclear magnetic resonance spectra ( $^1\text{H-NMR}$  and  $^{19}\text{F-NMR}$ ) and infrared resonance spectra (IR). With IR, "(neat)" represents measurement by liquid film and "(KBr)" represents measurement by molding a tablet. With NMR,  $\text{CDCl}_3$  represents the solvent, s a singlet, d a doublet, t a triplet, "sextet" a sextet, and m a multiplet. Also, for example, dt represents a double triplet, and "broad" represents broad absorption. The chemical shift is stated in units of ppm using tetramethylsilane ( $^1\text{H-NMR}$ ) and fluorotrichloromethane ( $^{19}\text{F-NMR}$ ) as internal standards and making the low magnetic field side a positive value.

[0050]

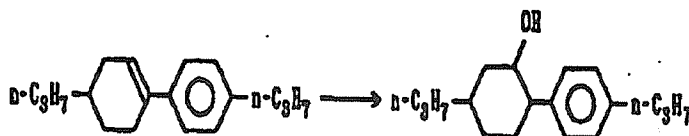
Practical Example 1

Synthesis of 2-fluoro-1-(4-propylphenyl)-4-propylcyclohexene (compound No. 1)

(1-a) Synthesis of 2-(4-propylphenyl)-5-propylcyclohexanol

[0051]

[Chemical formula 13]



[0052]

To a solution of 1.21 g (5 mmol) of 1-(4-propylphenyl)-4-propylcyclohexene in 10 mL of diethyl ether was added 2.5 mL (5 mmol) of a borane-dimethyl sulfide complex (2M ether solution) at room temperature, and this was stirred for 6 hr. The reaction solution was cooled to 0°C, and 5 mL of 3M sodium hydroxide was added and stirred for 10 min, following which 5 mL of 30% aqueous hydrogen peroxide was added. Next, the temperature was slowly elevated to room temperature, and after 1.5 hr the reaction solution was placed in a mixed solution of sodium hydrogen carbonate and sodium hydrogen sulfite, and the reaction product was extracted three times with 20 mL of diethyl ether. The organic phase was passed into a short column of anhydrous sodium sulfate and silica gel and concentrated, followed by purification using silica gel column chromatography to obtain 607 mg (2.35 mmol) of 2-(4-propylphenyl)-5-propylcyclohexanol. (yield: 47%)

[0053]

IR (neat) : 3460, 2962, 2932, 2872, 1720, 1516, 1466, 1382, 1340, 1260, 1250, 1113, 1050, 1020, 968, 908, 819, 738, 645 cm<sup>-1</sup>

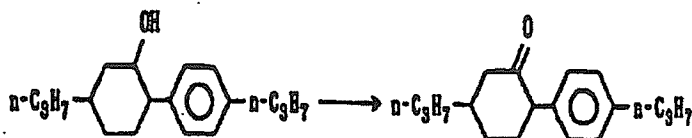
<sup>1</sup>H-NMR (CDCl<sub>3</sub>) : δ=0.92 (t, J=7 Hz, 6H), 1.1~2.0 (m, 13H), 2.0~2.6 (m, 2H), 2.60 (t, J=7 Hz, 2H), 3.5~4.0 (m, 1H), 7.16 (s, 4H)

[0054]

(1-b) Synthesis of 2-(4-propylphenyl)-5-propylcyclohexanone

[0055]

[Chemical formula 14]



[0056]

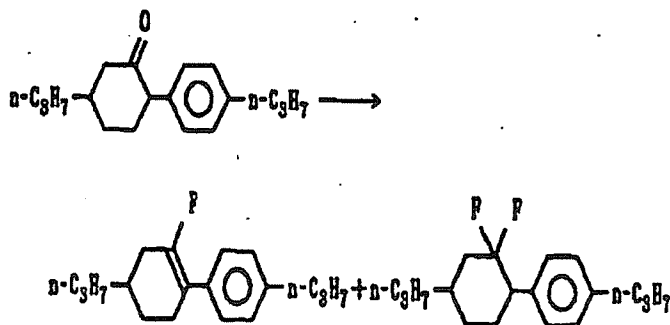
To a suspension of 10 mg (0.5 mmol) of pyridinium chlorochromate (PCC) in 1 mL of dichloromethane was added a solution of 65 mg (0.25 mmol) of 2-(4-propylphenyl)-5-propylcyclohexanol in 0.5 mL of dichloromethane at room temperature, and this was stirred for 2 hr. Then 5 mL of diethyl ether was added four times to the reaction solution, and the supernatant was collected and concentrated by passing it into a short column of anhydrous sodium sulfate and silica gel, followed by purification using silica gel thin-layer chromatography for fractionation to obtain 42 mg (0.16 mmol; yield: 65%) of 2-(4-propylphenyl)-5-propylcyclohexanone as an approximately 1:1 mixture of cis and trans forms and 3 mg (0.01 mmol; yield: 5%) of 1-(4-propylphenyl)-4-propylcyclohexene.

[0057]

(1-d) Synthesis of 2-fluoro-1-(4-propylphenyl)-4-propylcyclohexene

[0058]

[Chemical formula 15]



[0059]

To a solution of 130 mg (0.5 mmol) of an approximately 1:1 mixture of cis and trans forms of 2-(4-propylphenyl)-5-propylcyclohexanone obtained in (1-b) above in 1.5 mL of dichloromethane was added 0.20 mL (1.51 mmol) of diethylaminosulfur trifluoride (DAST), and

this was stirred for 11 hr. After the reaction concluded, 2 mL of diethyl ether and a saturated aqueous solution of sodium hydrogen carbonate were added, and after foaming stopped, 20 mL of water was added. The organic phase was concentrated by passing it into a short column of anhydrous sodium sulfate and silica gel, following which it was purified using silica gel thin-layer chromatography for fractionation to obtain 32 mg (0.12 mmol; yield: 24%) of 2-fluoro-1-(4-propylphenyl)-4-propylcyclohexene as an oily substance and 45 mg (0.16 mmol; yield: 32%) of *trans*-2,2-difluoro-1-(4-propylphenyl)-4-propylcyclohexane as an oily substance.

[0060]

#### 2-Fluoro-1-(4-propylphenyl)-4-propylcyclohexene

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ ) :  $\delta$ =0.94 (t, J=7.3 Hz, 6H), 1.3~1.5 (m, 5H), 1.64 (sextet, J=7.3 Hz, 6H), 1.7~1.9 (m, 2H), 1.9~2.1 (m, 1H), 2.4~2.5 (m, 3H), 2.57 (t, J=7.3 Hz, 2H), 7.14 (d, J=8.2 Hz, 2H), 7.33 (d, J=8.2 Hz, 2H)  
 $^{19}\text{F-NMR}$  ( $\text{CDCl}_3$ ) :  $\delta$ =-105.08 (s)

[0061]

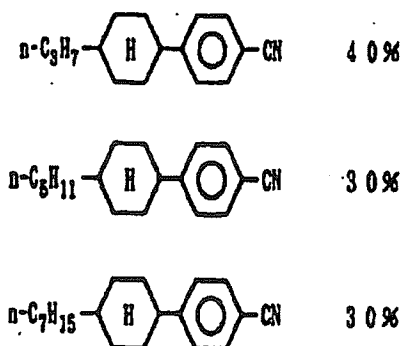
#### Practical Example 2

##### Preparation of liquid crystal composition

When phenylcyclohexane parent liquid crystals (A) of the following composition

[0062]

[Chemical formula 16]



[0063]

(the cyclohexane ring represents the *trans* configuration) were prepared, they showed a nematic

(N) phase at  $\leq 50.5^{\circ}\text{C}$ . Their physical properties and the threshold voltage ( $V_{th}$ ) of a TN cells produced using them were as follow.

[0064]

Dielectric anisotropy ( $\Delta\epsilon$ ): 12.0  
 Refractive index anisotropy ( $\Delta n$ ): 0.117  
 Threshold voltage ( $V_{th}$ ): 1.50 V

[0065]

A liquid crystal composition comprising 90% of these parent liquid crystals (A) and 10% of compound No. 1 obtained in Practical Example 1 was prepared. The upper-limit temperature of the N phase ( $T_{N-I}$ ) of this liquid crystal composition (M-1) and its physical properties were as follow.

[0066]

Upper-limit temperature of N phase ( $T_{N-I}$ ):  $40.6^{\circ}\text{C}$   
 Dielectric anisotropy ( $\Delta\epsilon$ ): 9.8  
 Refractive index anisotropy ( $\Delta n$ ): 0.107  
 Threshold voltage ( $V_{th}$ ): 1.26 V

[0067]

Thus it is clear that in spite of the fact that its dielectric anisotropy ( $\Delta\epsilon$ ) was smaller, its threshold voltage was extremely low. In view of equation (1) above, this indicates that the elastic constant (K) of compound No. 1 of this invention is extremely low. It is also clear that its refractive index anisotropy ( $\Delta n$ ) is considerably smaller.

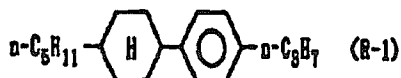
[0068]

#### Comparative Example 1

Liquid crystal composition (N-1) comprising 10% of the compound of formula (R-1), a compound that has been used for the same purpose in the past, instead of compound No. 1 used in Practical Example 2

[0069]

[Chemical formula 17]



[0070]

and 90% of parent liquid crystals (A) was prepared. The upper-limit temperature of the N phase ( $T_{N-I}$ ) of this liquid crystal composition (N-1) and its physical properties were as follow.

[0071]

Upper-limit temperature of N phase ( $T_{N-I}$ ): 39.9°C

Dielectric anisotropy ( $\Delta\epsilon$ ): 11.3

Refractive index anisotropy ( $\Delta n$ ): 0.109

Threshold voltage ( $V_{th}$ ): 1.37 V

[0072]

Thus the upper-limit temperature of the N phase was 39.9°C, virtually unchanged from that of liquid crystal composition (M-1), but in spite of the fact that its dielectric anisotropy ( $\Delta\epsilon$ ) was larger than that of liquid crystal composition (M-1), its threshold voltage ( $V_{th}$ ) was considerably higher than that of liquid crystal composition (M-1). Its refractive index anisotropy ( $\Delta n$ ) was also somewhat larger than that of liquid crystal composition (M-1).

[0073]

It is clear from these results that adding a small amount of the compound of general formula (I) to matrix crystals (A) can very effectively lower the threshold voltage ( $V_{th}$ ) of the parent crystals and also has the effect of lowering  $\Delta n$ .

[0074]

#### Effects of the invention

The compounds represented by general formula (I) of this invention are outstanding in compatibility with the parent liquid crystals commonly used at present as nematic liquid crystals, and adding a small amount can effectively lower the threshold voltage ( $V_{th}$ ) of liquid crystal cells produced using these in spite of the fact that the dielectric anisotropy ( $\Delta\epsilon$ ) becomes smaller, so they are useful as materials for various liquid display elements. They can also make the refractive index anisotropy ( $\Delta n$ ) smaller, so they are ideal as materials for constructing liquid crystal display elements with a wide-angle field of view.

[0075]

Also, the compounds of general formula (I) can be easily produced industrially, as shown in the practical examples, and they are chemically stably to heat, light, and water, so they are very



useful as materials for various liquid crystal display elements.

[Translator's note: The information on the last page that was continued from the front page has been incorporated into the translation.]